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Evaluation of dry heat treatment of soft wheat flour for the production of high ratio cakes

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ABSTRACT

An accurate method to heat treat flour samples has been used to quantify the effects of heat treatment on flour functionality. A variety of analytical methods has been used such as oscillatory rheology, rheomixer, solvent retention capacity tests, and Rapid Visco Analysis (RVA) in water and in aqueous solutions of sucrose, lactic acid, and sodium carbonate. This work supports the hypothesis that heat treatment facilitates the swelling of starch granules at elevated temperature. Results furthermore indicated improved swelling ability and increased interactions of flour polymers (in particular arabinoxylans) of heat treated flour at ambient conditions. The significant denaturation of the proteins was indicated by a lack of gluten network formation after severe heat treatments as shown by rheomixer traces. Results of these analyses were used to develop a possible cake flour specification. A method was developed using response surfaces of heat treated flour samples in the RVA using i) water and ii) 50% sucrose solution. This can uniquely characterise the heat treatment a flour sample has received and to establish a cake flour specification. This approach might be useful for the characterisation of processed samples, rather than by baking cakes. Hence, it may no longer be needed to bake a cake after flour heat treatment to assess the suitability of the flour for high ratio cake production, but 2 types of RVA tests suffice.

1. Introduction

The production of traditional cakes such as pound cakes requires the use of equal amounts of sugar, flour, eggs, and fat. These cakes exhibit a coarse texture and a limited shelf life (Catterall, 2000). Montzheimer (1931) discovered that chlorine gas improved the baking performance of flour, making it more tolerant to high sugar concentrations in the formulation (Guy & Pithawala, 1981). Thereafter, high ratio cakes with sugar to flour ratios of 1.0–1.4 were developed. These cakes exhibit a higher level of sweetness, a moist mouthfeel, a fine texture, and a long shelf life. Hence, high ratio cake formulations such as sponges, cupcakes, or gateaux cakes are widespread in industry and have proved very successful in the packaged cake market due to their good eating and keeping qualities (Guy & Pithawala, 1981; Hodge, 1975).

The production of successful high ratio cakes requires the use of cake flour. Cake flour is flour which has been treated to alter its functionality to carry more water and sugar leading to the desired cake quality attributes (Collyer, 1968; Cook, 2002). The most effective method for this purpose is to expose flour to chlorine gas (Bent, Bennion, & Bamford, 1997). The dry heat treatment of flour is an alternative process to chlorination and is the focus of this study.

Industrially, the dry heat treatment of flour typically involves several processing steps such as drying of flour in a stream of hot air, heat treatment via contact heating, cooling, rehydration, and milling for size reduction (Chesterton, Wilson, Sadd, & Moggridge, 2015). Heat treatment commonly uses rotating drums or heated conveyors (Guy & Mair, 1990; Nicholas, Boynton, Russo, & Totty, 1978). A novel device for the continuous heat treatment of flour was described by Keppler, Bakalis, Leadley, and Fryer (2016a).

Mechanisms for the improvement of baking performance of dry heat treated flour are unclear (Chesterton et al., 2015). However, it is widely accepted that starch properties are affected, in that starch granule swelling is facilitated allowing for development of the desired cake structure (Gough, Greenwood, & Whitehouse, 1977; Guy & Pithawala, 1981; Guy, Skinner, & Sahi, 2007). During heating of the batter, swollen starch granules form a rigid gel-like structure by intergranular contacts surrounded by agglomerated protein. These allow previously introduced air cells in the cake batter to burst, creating a stable and open structure (Guy et al., 2007; Guy & Pithawala, 1981). If starch granules do not swell sufficiently, air cells do not rupture, but shrink during cooling and the cake collapses. The modification of starch functionality has been attributed to changes in the surface layers (e.g.

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protein and lipids) of the granules (Cook, 2002).

Assessment of the success of any treatment is largely achieved by characterising the quality of a baked cake (Neill, Al-Muhtaseb, & Magee, 2012; Thomasson, Miller, & Hoseney, 1995). Evaluation of the treated flour with a simple analytical method would be preferable. However, there is no simple analytical method to test flour for its suitability in high ratio cakes.

The focus of this study is the dry heat treatment of flour and the aims are to

i) gain a better understanding of the dry heat treatment of flour and to quantify the effects of heat on flour functionality. Heat treated flour is analysed by a variety of analytical methods such as the Rapid-Visco-Analyser (RVA), Solvent retention capacity (SRC) tests, the rheometer, and the rheomixer.
ii) develop a cake flour specification using the obtained data by RVA, SRC test, the rheometer, and the rheomixer.

2. Material and methods

2.1. Material

Commercially available soft wheat flour (Golden Dawn flour, supplied by ADM Milling Ltd. (Chelmsford, UK)) was used for the experiments. It contained 8.8% protein and 12.5% moisture. Further analyses done at Campden BRI showed a damaged starch level of 23 FU and a Hagberg falling number of 330 s.

2.2. Methods

2.2.1. Heat treatment

Preliminary experiments showed that when a flour layer 0.5–1.0 cm thick was heated in a convection oven set at 150 °C, the flour temperature takes approx. 10 min to reach the setpoint. This is due to a low thermal conductivity of flour (e.g. 0.17 W/mK) and a low heat transfer coefficient of air in the oven (Tiwari, Wang, Tang, & Birla, 2011). Here, an accurate method of heat treatment was developed which ensured rapid heating and a constant temperature of the flour throughout the treatment. This was done by sieving flour (7 ± 0.2 g) in a thin layer of 1 mm onto a quadratic hotplate (Stuart SD 162) with an area of 234 cm^2. The mechanism of heat transfer is conduction, giving results comparable to an industrial device for the heat treatment of flour described by Keppler et al. (2016a).

The temperature on the surface of the hotplate was monitored by k-type thermocouples. Small variation in temperature was observed in the initial phase of loading and heating the sample (< 5 °C), but after 2–3 min, the temperature profile was constant with time. An accurate heat treatment was achieved with this method. Typically, heat was applied at temperatures between 110 °C and 200 °C for between 1 and 30 min (see Table 2).

2.2.2. Rapid-Visco-Analyser (RVA) tests

Starch pasting behaviour was studied by Rapid Visco Analysis (RVA 4500, Perten Instruments (Hägersten, Sweden)). In the sample canister, flour samples (3.0 g dm) were added to 25 g of different liquids: (i) deionized water, (ii) 50% w/w sucrose in water, (iii) 5% w/w lactic acid in water and (iv) 5% w/w sodium carbonate in water. The effect of varying moisture contents of the samples was compensated for by added deionized water. The sucrose solution was prepared at least 12 h in advance to ensure complete dissolution. Coated cans were used for the 5% w/w sodium carbonate solution to prevent corrosion of the aluminium canisters. The temperature profile applied was Standard 1 method (AACCI Approved Method 76-21.01, 1999). Pasting temperature, peak viscosity, holding strength, setback, and final viscosity were calculated from the data (ThermoCline for Windows version 3).

2.2.3. Solvent retention capacity

Solvent retention capacity (SRC) tests were performed according to AACCI Approved Method 56-11.02 (2009). The SRC test is a solvation assay for flours based on the enhanced swelling behaviour of individual polymer networks in selected single diagnostic solvents (Kweon et al., 2009). The solvents were deionized water, 5% w/w lactic acid in water, 5% w/w sodium carbonate in water, 50% w/w sucrose in water.

Flour (5 ± 0.050 g) was mixed with 25 g of solvent in 50 ml centrifuge tubes. The flour was then left to solvate for 20 min during which the tubes were vigorously shaken every 5 min. The samples were centrifuged at 1000 x g for 15 min. The supernatant was discarded and the tubes were left upside down to dry for 10 min. The pellet was weighed and the SRC was calculated by Eq. (1).

$$\%\mathrm{SRC} = \left[\frac{\text{pellet wt}}{\text{flour wt}} - 1\right]\cdot\left[\frac{86}{100 - \%\text{flour moisture}}\right]\cdot 100 \quad (1)$$

2.2.4. Small deformation oscillatory measurements

The viscoelastic properties of flour-water slurries were studied at 20 °C on a rheometer (Ares, type 902-30004 (Rheometric Scientific Ltd., Hampshire, UK)) using a parallel plate setup (50 mm diameter and 2 mm gap).

Flour (15 ± 0.1 g, 14% moisture) was mixed with 20 g of deionized water manually with a spatula for 1 min. As with RVA tests, sample mass was corrected for varying moisture contents of the heat treated flour. To exclude the effect of time, all samples were loaded onto the rheometer 3 min after mixing. After loading, the edges of the sample were trimmed with a spatula and the sample held for 1 min. Strain sweeps were performed (frequency of 1 Hz and strain range from 0.007 to 188.8%) to identify the linear viscoelastic region. A strain of 0.1% was imposed and frequency sweeps conducted between 0.1 Hz and 20 Hz. The elastic moduli at a frequency of 5 Hz were compared.

2.2.5. Rheomixer

Flour samples were subjected to torque-time analysis in the rheomixer to compare their dough development and protein quality. The instrument (Reologica Instruments AB, Lund, Sweden) consists of an instrumented planetary pin mixer, a microprocessor unit for signal conditions and software for data acquisition. It is described in Anderson (2003) and is designed to be similar to a mixograph (AACCI Approved Method 54-40.02, 1999). The rheomixer has a removable bowl (for 10 g flour) that has 3 fixed pins and is located in a water jacket to allow constant temperature mixing. Mixing action is provided by 2 pairs of driven pins that are lowered into the bowl and are set rotating. When dough is mixed in the apparatus, a torque is imparted to the bowl which is recorded as a voltage. The primary output is a torque-time trace.

Flour (10 ± 0.1 g) was added into a tempered (30 °C) rheomixer bowl, which was then covered with Parafilm and put into a water bath at 30 °C for at least 5 min. Three grooves were made into the flour which connected in the middle of the bowl where 9 g of dist. water was added. The bowl was then transferred into the rheomixer where the dough was developed at a speed of 93 ± 2 rpm for 10 min.

2.2.6. Statistical methods

Heat treatment of flour for analysis in the RVA was performed in triplicate in preliminary experiments. Results demonstrated repeatable heat treatment as shown by small standard deviation of the measured parameter (peak viscosity) of ± 1%. Here, individual experiments were done without replicates to maximise the number of studies possible from the same sample.

Regression analysis was performed in MATLAB 8.2 and Microsoft Excel 2013. Response surfaces and contour plots were created in MATLAB 8.2.

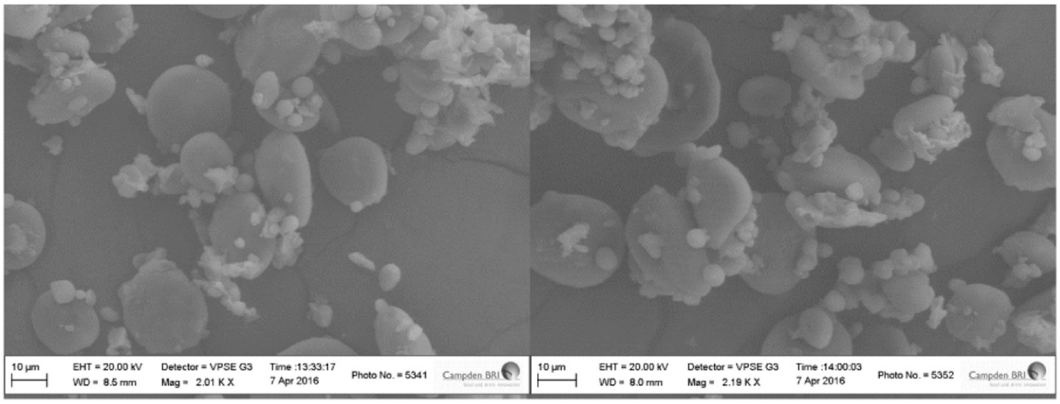


Fig. 1. SEM micrographs of untreated flour (left) and heat treated flour (170 °C, 15 min) (right).

3. Results and discussion

Preliminary scanning electron microscope (SEM) studies (see Fig. 1) of untreated and heat treated flour samples showed no difference between the appearance of starch granules and surrounding protein, in agreement with Russo and Doe (1970). Hence, the changes in flour functionality caused by heat treatment discussed below do not result from changes in the morphology of flour components.

Table 1 lists the methods used here to characterise and quantify the effects of heat treatment on flour functionality. The obtained results are used in an attempt to develop a cake flour specification.

3.1. Rapid-Visco-Analyser tests in different fluids

Starch granules can absorb up to 30% of their dry weight of water and swell thereby slightly (Manley, Pareyt, & Delcour, 2011). When the starch suspension is heated above the so called gelatinisation temperature, the molecular order of the granule is irreversibly destroyed (Manley et al., 2011; Ross, 2012). The events that occur following starch gelatinisation during further heating are referred to as pasting (Manley et al., 2011; Ross, 2012). The starch pasting behaviour of heat treated flour samples in different solutions in the Rapid-Visco-Analyser is discussed below.

3.1.1. In water

The pasting behaviour of starch is evaluated in the RVA. A typical RVA profile for flour in water is shown in Fig. 2. Parameters like pasting temperature, peak viscosity, holding strength, setback, and final viscosity can be calculated. The peak viscosity is used here to assess the effect of heat treatment on flour. It is a measure of the viscosity and the combination of two processes: i) granule swelling and polymer leaching, which increases viscosity and ii) granule rupture and polymer alignment which decreases it (Crosbie & Ross, 2007).

Fig. 3 shows the effect of treatment time and temperature on the peak viscosity of heat treated flour samples. At all temperatures measured, the peak viscosity first increases with treatment time before it reaches a maximum and then declines. The maximum is reached at shorter times for higher process temperatures: i.e. 2 min at 170 °C against 12 min at 150 °C. The figure shows treatment times up to 30 min, but the decreasing trend of peak viscosity for long times and lower temperatures (e.g. 110 °C and 130 °C) was confirmed in preliminary experiments (see Fig. 1, Supplementary material).

The increase in peak viscosity over time suggests either (i) granule swelling is facilitated by heat treatment or (ii) granule disruption by external shear is delayed giving the granules more time to swell. Nicholas et al. (1978) stated that starch granules swell more easily in heat treated samples due to partial denaturation of the proteins on the granules surface which are thought to form a barrier hindering water absorption into the granule (Cook, 2002; Guy & Pithawala, 1981). Van Steertegem et al. (2013) reported that the peak viscosity increased upon longer and more severe heat treatment due to covalent disulphide cross-links in the gluten proteins. The polymerised protein rigidifies the flour particle and allows it to swell longer and to withstand external shear such that the RVA peak viscosity increases (Van Steertegem et al., 2013).

The decreasing peak viscosity after a certain treatment time indicates less swelling of the starch granules. This might be due to fragmentation of starch or due to changes in the granule surface by extreme heat treatment that prevent or delay water uptake. Furthermore, the starch granules might be more susceptible to shear. Earlier granule rupture and polymer alignment might result in a low peak viscosity.

After interpolation of all the data, Fig. 4a shows the response surface for the peak viscosity of heat treated flour samples as a function of treatment time and temperature.

3.1.2. In 50% sucrose solution

To distinguish between treatment conditions it is preferable to obtain large differences in the observed parameter (here: peak viscosity) after various heat treatments. Kweon, Slade, Levine, and Souza (2010) reported a greater difference in the RVA profiles for different levels of flour chlorination when 50% sucrose solution was used instead of water. This phenomenon was therefore investigated for heat treated samples as presented below.

The effect of treatment time at 150 °C on the RVA traces in 50% sucrose solution is shown in Fig. 5a. The profile shape is significantly different from that in water (Fig. 2). In agreement with Kweon et al. (2010), the profile shows that starch breakdown is negligible in sucrose solution, i.e. the traces do not decrease with time (compare Fig. 2 and

Table 1
Analytical methods used to characterise and quantify the effects of heat treatment on flour functionality.

Analytical method	Measurement of	Quantification parameter/of
Rapid-Visco-Analyser tests in 4 solutions	Pasting behaviour of starch	Peak viscosity
Small deformation oscillatory measurements	Viscoelastic behaviour of flour-water slurry	Elastic modulus
Solvent retention capacity tests	Swelling behaviour of flour polymers in different solvents	% SRC
Rheomixer	Dough development	Mixing curves

Table 2
Time-temperature combinations of dry heat treatment of flour on hot plate.

Temperature [°C]	RVA in water	RVA in 50% sucrose solution	RVA in 5% lactic acid solution	RVA in 5% sodium carbonate solution	Rheological measurements	SRC	Rheomixer
110	5, 10, 15, 20, 30 min	2, 5, 10, 20, 30 min	–	10, 20 min	5, 10, 15 min	5, 10, 15, 20 min	–
130	1, 3, 5, 10, 15, 17, 20, 33 min	2, 5, 20, 30 min	–	10 min	10, 30, 40, 60 min	5, 10, 15, 20 min	20, 30 min
135	–	–	–	–	–	–	5 min
140	–	–	–	–	–	–	5 min
150	1, 3, 5, 7, 10, 12, 15, 20, 25, 30 min	2, 5, 10, 15, 20 min	–	–	5, 10, 20 min	2, 5, 10, 15, 20 min	–
160	1, 3, 5, 7, 10, 11, 13, 15, 25 min	–	–	–	–	–	5 min
170	1, 2, 3, 4, 5, 7, 8, 10, 15, 30 min	5, 10, 15, 20, 30 min	2, 4, 6, 8, 10 min	–	1, 5, 10, 30 min	2, 4, 6, 8, 15 min	1, 5, 15 min
190	–	–	–	–	1, 2, 5 min	1, 2, 4, 6, 8, 10 min	–
200	–	–	–	–	1, 2, 5 min	–	–

Fig. 5a). Kweon et al. (2010) suggest viscosity development in the second-stage granule-swelling process is retarded and hence there is insufficient time for the subsequent disruption process, before cooling and setback.

In comparison to the RVA using water (see Fig. 4a), there is a greater difference between investigated conditions. This suggests that the mechanism that increases peak viscosity in water is more efficient in sucrose. Sucrose might stabilise the starch granules against disruption, thereby increasing the peak viscosity. Kweon et al. (2010) suggests that swelling of arabinoxylans in solvent retention capacity (SRC) tests, also observed here for heat treated samples in this study, could be responsible for the exaggerating effect in the RVA profiles.

As there is no maximum, the "peak" viscosity is taken during the holding time at 95 °C at 7 min, and this is shown in Fig. 5b as a function of process time and temperature. The peak viscosity increased linearly with time at all temperatures. This is significantly different from the results from the RVA in water, where a maximum peak viscosity was reached after a specific treatment time. Additionally, the initial peak viscosity is lower in 50% sucrose solution than it is in water suggesting that starch granule swelling is facilitated in water. However, the overall increase in peak viscosity after heat treatment is higher in 50% sucrose solution than in water indicating a higher potential for granule swelling after heat treatment.

The response surface for peak viscosity of flour samples treated at

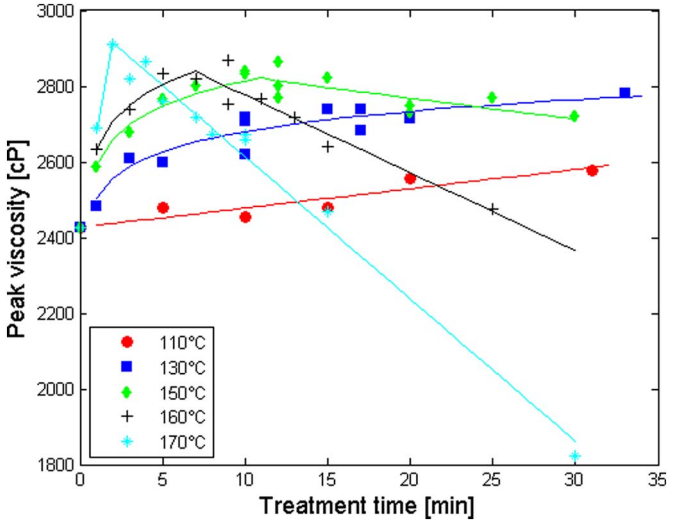


Fig. 3. Effect of treatment time and temperature on the RVA peak viscosity of flour in water (with fitted exponential and straight lines).

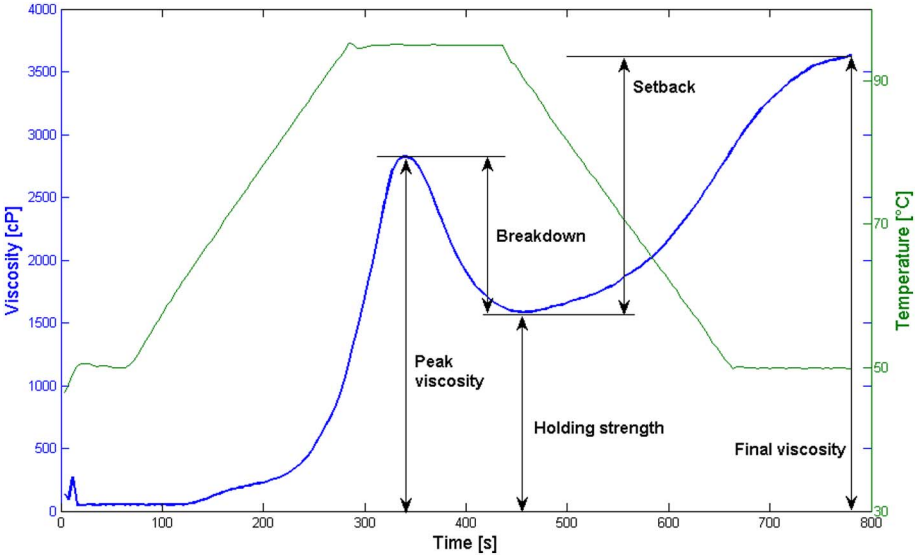


Fig. 2. Typical Rapid Visco Analysis (RVA) profile of heat treated flour (150 °C, 15 min) in water.

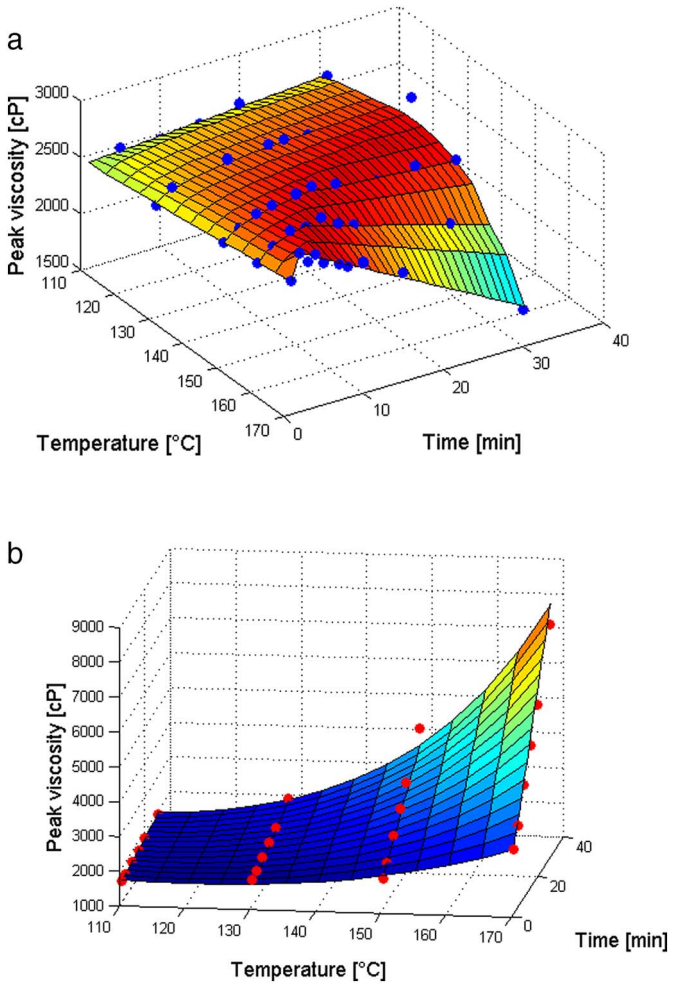


Fig. 4. Response surfaces of the RVA peak viscosity of flour treated at different treatment times and temperatures: a) in water b) in 50% sucrose solution. Dots denote experimental data.

different time-temperature combinations is presented in Fig. 4b; Fig. 4a and b can be compared to show the differences between water and sucrose solutions. In water the lowest value is obtained at high temperatures and long times, whereas in 50% sucrose solution the lowest value is at low temperatures and short times.

3.1.3. In 5% lactic acid solution and in 5% sodium carbonate solution

Fig. 5c shows the RVA profile for flour treated at 170 °C for different times in 5% lactic acid solution.

The shape of the trace is similar to that for water, with a marked peak viscosity followed by a trough and an increase to a final viscosity. The profile shifts to the left with increasing treatment intensity indicating that the swelling process of the starch granules is facilitated. The holding strength is significantly lower in lactic acid solution compared with water. The breakdown viscosity (difference between peak viscosity and holding strength) lies between 950 cP and 1190 cP for heat treated flour (170 °C for 2–10 min) in water, whilst it is 2030–2520 cP in lactic acid. This suggests that the disruption of the starch granules (starch breakdown) is more severe in the acid.

In contrast to profiles in water, but in agreement with those in 50% sucrose solution, the peak viscosity increased linearly with treatment time (see Fig. 2, Supplementary material). The results indicate that the mechanism destabilising the system in water (no gluten network formation and hence no stabilisation against shear) is balanced or compensated for by sucrose or lactic acid.

The rate at which granules were disrupted after the peak viscosity was reached was identical at all treatment times, as shown by the overlapping curves. Moreover, the peak viscosity for all treatment times lies on an extended straight line of the decreasing part of all curves.

The RVA test in 5% sodium carbonate solution showed no difference between the traces of untreated and heat treated flour samples. The findings suggest either that

i) changes in flour that occur during heat treatment are reversed by 5% sodium carbonate solution, or
ii) changes induced by heat treatment can also be achieved by the alkaline environment in a short time. Thereby, all transformations are complete in the RVA solution causing equivalent traces; or
iii) the RVA test in sodium carbonate is insensitive to changes induced by heat treatment.

3.2. Solvent retention capacity

The SRC test is a solvation assay for flours based on the enhanced swelling behaviour of individual polymer networks in selected single diagnostic solvents. These are used to predict the functional contribution of each individual flour component (Kweon, Slade, & Levine, 2011). Water is used to evaluate the overall swelling behaviour of all network-forming components; 5% lactic acid SRC is related to glutenin network forming and gluten strength; 5% sodium carbonate favours swelling behaviour of damaged starch; and sucrose SRC assesses the swelling behaviour of water-accessible arabinoxylans (Kweon et al., 2009).

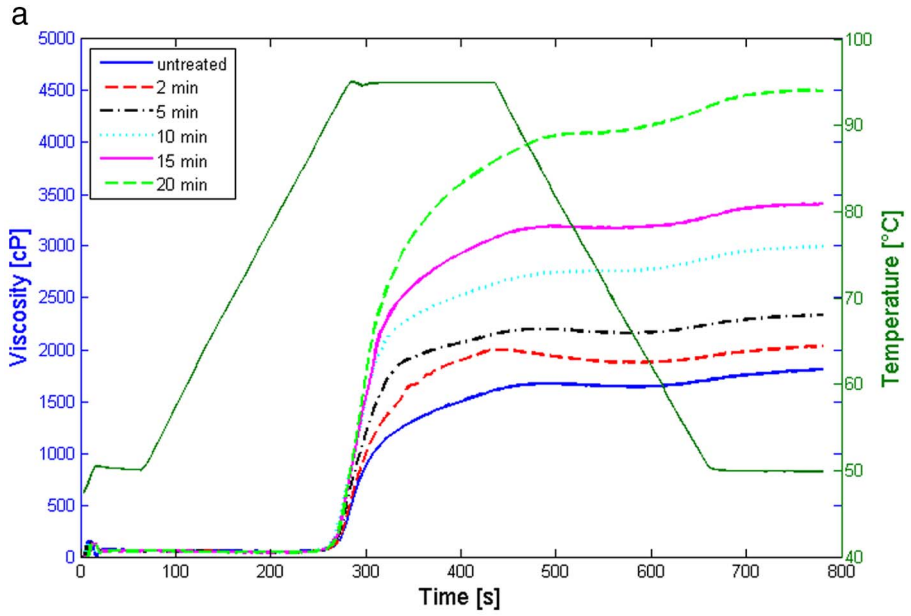
Fig. 6a compares the SRC for all four solvents for samples treated at 150 °C for different times. The water SRC increases over time, indicating the improvement of swelling ability of the flour polymers, in agreement with Van Steertegem et al. (2013). Lactic acid SRC increased up to 5 min treatment time and decreased slowly after that, in contrast to Van Steertegem et al. (2013) who reported decreasing lactic acid SRC indicating a decreased swelling ability of the gluten network. As with lactic acid, the sodium carbonate SRC increased up to a 5 min treatment time, after which it changed only marginally indicating the limited effect of heat treatment on swelling behaviour of damaged starch (Kweon et al., 2009; Van Steertegem et al., 2013). The biggest impact of heat treatment was observed in sucrose SRC, which increased by approximately 40%. Van Steertegem et al. (2013) suggested that the solvent accessibility of arabinoxylans was impacted by heat treatment. A drastic increase in sucrose SRC was also reported for chlorinated flour (Kweon et al., 2009).

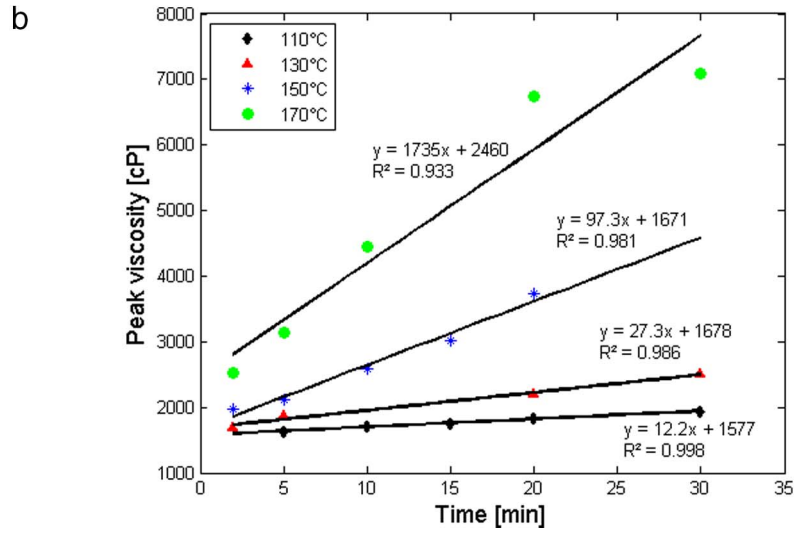
The Gluten Performance Index (see Eq. (2)) described by Kweon et al. (2009) as a better predictor of gluten functionality than lactic acid SRC alone is also shown in Fig. 6a. It describes the overall performance capability of glutenin in the environment of other modulating networks (Kweon et al., 2011), and it is defined as:

$$\text{GPI} = \frac{\text{Lactic acid SRC}}{\text{Sodium carbonate SRC} + \text{Sucrose SRC}} \quad (2)$$

The GPI increased up to 5 min, then decreased with increasing treatment time. This is mostly due to the significant increase of sucrose SRC rather than with a decrease in lactic acid SRC. Results suggest that there is an optimum heat treatment time for optimum gluten network formation and protein hydration.

The water SRC for different treatment times and temperatures is shown in Fig. 6b. It linearly increases with time for all temperatures showing the improvement of swelling ability of the flour polymers with increasing treatment intensity. This may cause larger contact areas and increased interactions of the various flour components after heat treatment. These findings agree with the elastic behaviour of heat treated flour in the rheological study below. The slope of the increasing water SRC with treatment time increased exponentially with increasing treatment temperature (see Fig. 3, Supplementary material).





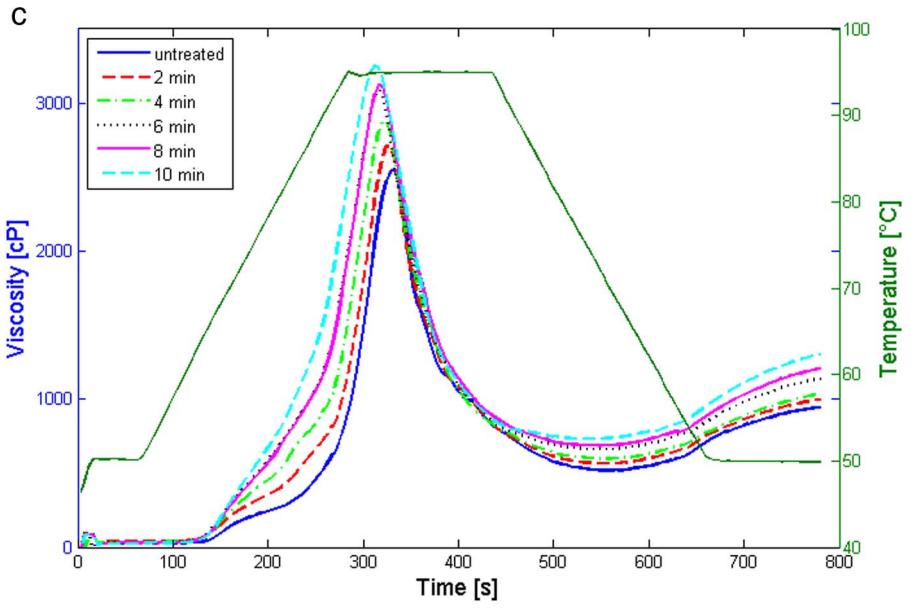


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Fig. 5. a) RVA profiles in 50% sucrose solution of flour treated at 150 °C for different times. b) RVA peak viscosity in 50% sucrose solution of heat treated flour samples (110–170 °C, 2–30 min); with best-fit lines. c) RVA profiles in 5% lactic acid solution of heat treated flour samples (170 °C, 2–10 min).

3.3. Small deformation oscillatory measurements

Rheological measurements evaluate the viscoelastic behaviour of a flour-water slurry. The storage modulus relates to the elastic component indicating the amount of energy stored. The elastic flow characterises a reversible deformation. The loss modulus relates to the viscous component indicating the amount of energy dissipated through generated heat (Miri, 2010). Viscous flow characterises irreversible deformation.

Fig. 7 shows the effect of treatment time (150 °C) on the storage and loss moduli. The elastic component dominates, and both moduli increase with increasing treatment time. Flour samples treated between 110 °C and 200 °C for 5 min display significantly higher storage moduli than loss moduli in all cases (see Fig. 4, Supplementary material). Both moduli increase with increasing temperature.

The time and temperature dependence of the elastic modulus is shown in Fig. 8a and as a response surface in Fig. 8b. Elastic behaviour increased linearly with time for all temperatures. The slope of the increase in elastic modules with increasing treatment time (= reaction rate) increased exponentially with temperature (see Fig. 5, Supplementary material). When the logarithm of this reaction rate is plotted against the reciprocal temperature, an activation energy of −96.4 kJ/mol is obtained.

The results suggest that more severe heat treatments cause increased interactions of flour components, so that the system is can store more energy. Possible explanations include:

i) The proteins on the starch granule surface form a barrier and hinder water absorption into the untreated granule (Nicholas et al., 1978). The barrier might be degraded by partial denaturation of the proteins allowing the granules to swell more easily at ambient temperature. Increased swelling of polymers might allow for more contact areas, interactions, and the ability to store more energy.
ii) Heat treatment impacts on the arabinoxylan fraction of flour as suggested by Van Steertegem et al. (2013). It is this component

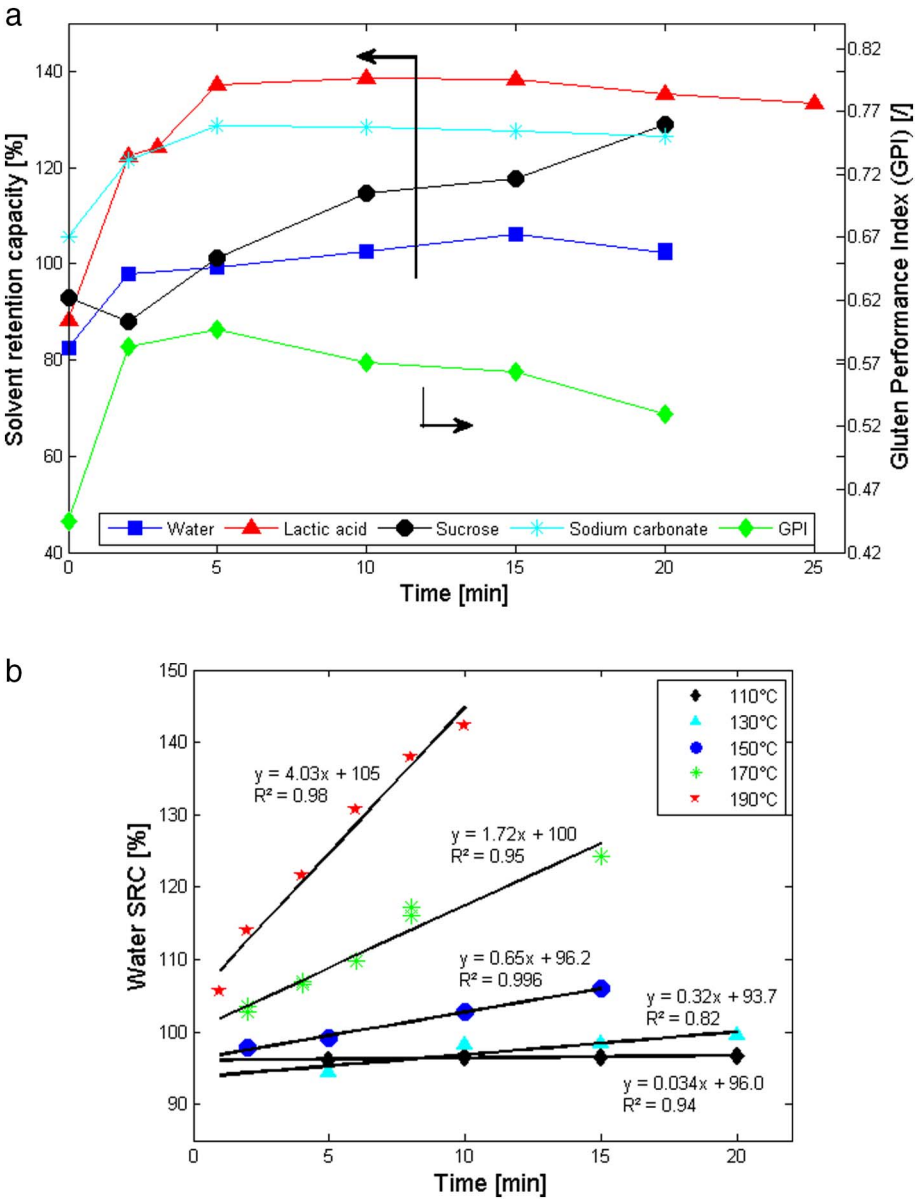

Fig. 6. a) Solvent retention capacity (SRC) of flour samples treated at 150 °C for different times. b) Water SRC of flour samples treated at different temperatures for different times.

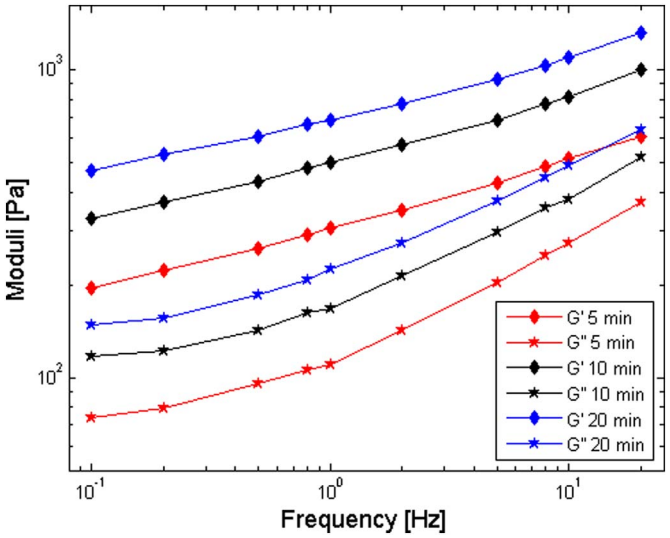

Fig. 7. Effect of heat treatment time (at 150 °C) on elastic (G′) and viscous (G″) moduli of a flour-water slurry.

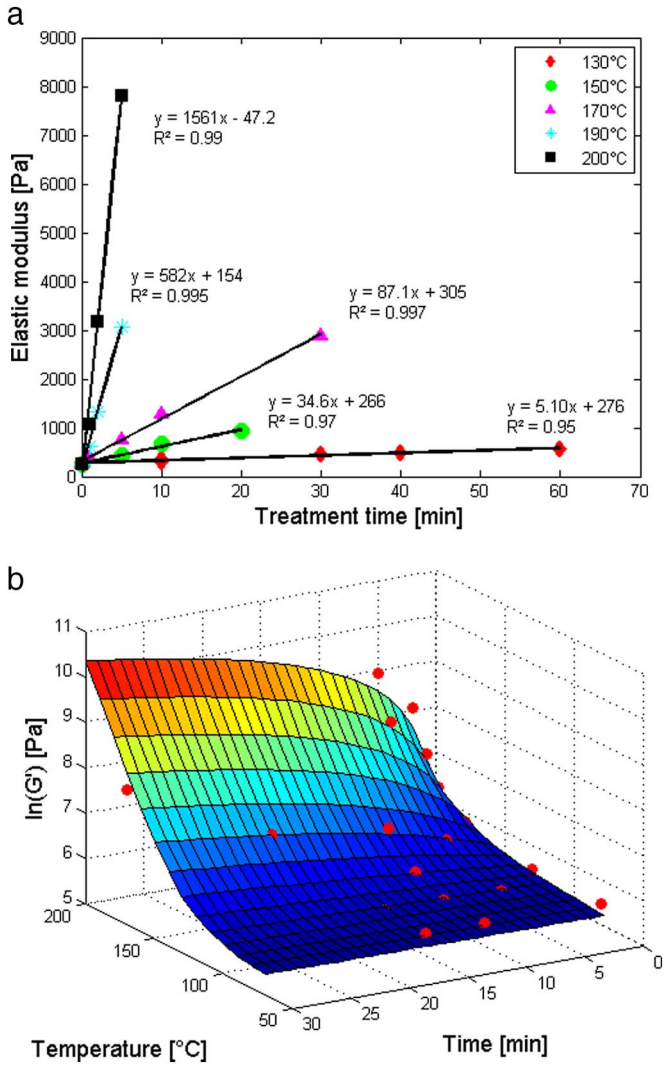

Fig. 8. a) Elastic modulus of heat treated flour samples (130–200 °C, 1–60 min) in water b) Response surface of elastic modulus of heat treated flour samples in water as a function of treatment temperature and time with data points (R^2 = 0.85).

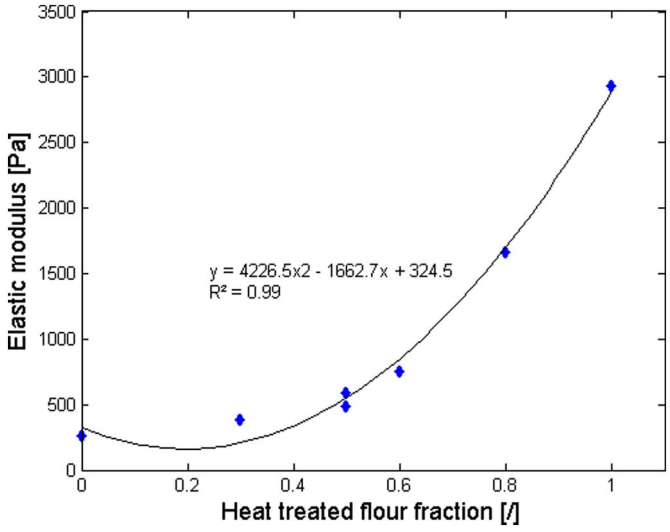

Fig. 9. Elastic modulus of untreated flour, heat treated flour (190 °C for 5 min), and combinations; with best-fit line.

whose swelling behaviour in the SRC test increases most upon heat treatment.

In continuous heating, the product has a process specific residence time distribution as it passes through the equipment (Keppler, Bakalis, Leadley, & Fryer, 2016b). To evaluate the impact of the width of the residence time distribution on flour functionality, it is important to understand the changes in functionality of flour mixtures containing flours that were treated for different times. For that reason, untreated flour was combined with heat treated flour (190 °C for 5 min) and the elastic modulus was measured.

Fig. 9 shows the elastic modulus G′ of untreated flour (heat treated flour fraction = 0), heat treated flour, and various combinations. The elastic modulus is not linear as a function of the heat treated flour fraction suggesting the untreated flour is the dominant component.

3.4. Rheomixer

Gluten proteins are responsible for the unique viscoelastic properties of wheat dough (Mann, Schiedt, Baumann, Conde-Petit, & Vilgis, 2014). Their three-dimensional structure unfolds upon mixing and water addition and a transient network is formed, comprised mainly of disulphide bonds, hydrogen bonds, hydrophobic interactions and entanglements (Mann et al., 2014). Thermal treatments can alter protein conformation, their ability to take part in these processes and hence, affect the dough formation process (Mann et al., 2014).

Fig. 10 shows the mixing curves in the rheomixer of flour samples treated at various temperatures for 5 min:

- In the untreated sample the gluten is hydrated and a network is developed upon mixing. The curves of the flour samples treated up to 135 °C look similar suggesting a gluten network is formed.
- The curves for samples treated between 140 °C and 160 °C are marked by a significant drop in mixing torque after 5–8 min of mixing time indicating the instability of the dough and its sensitivity to mechanical treatment.
- The profiles at 170 °C and 180 °C (not shown) show no drop in mixing torque indicating a stable dough. However, the shape of the mixing curve is different from those at lower temperatures. The maximum torque is lower and the curve is not increasing, but constant with mixing time.

Van Steertegem et al. (2013) reported an impact of heat treatment

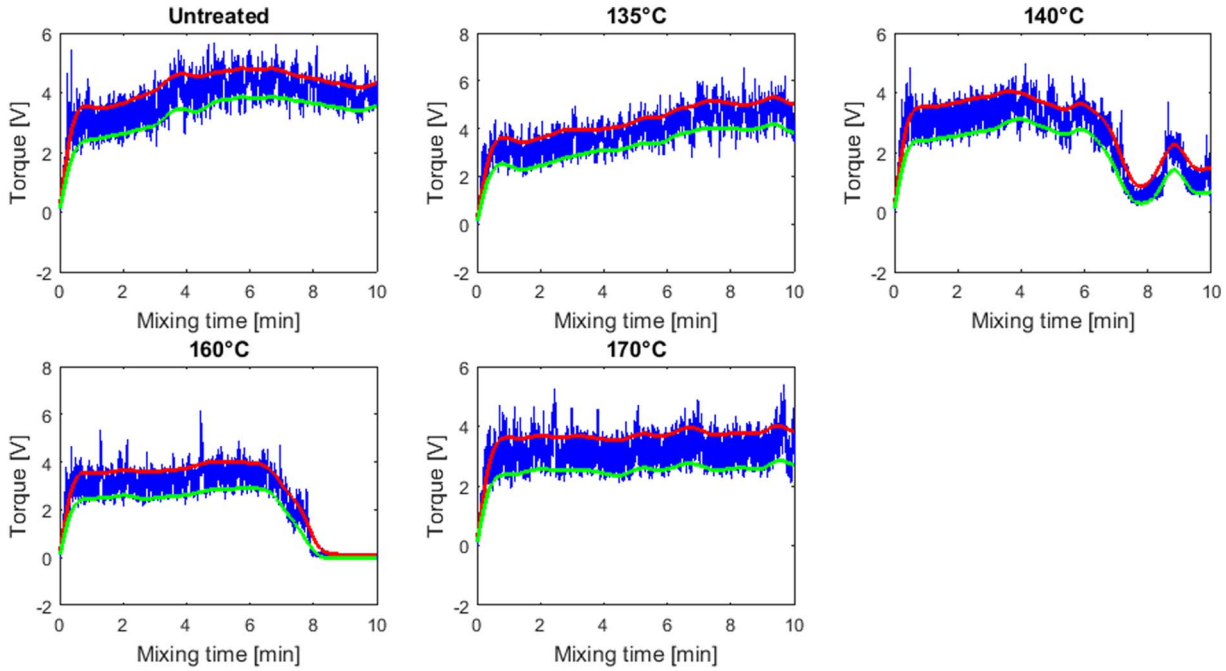

Fig. 10. Mixing curves of flour samples treated at different temperatures for 5 min; red and green lines show upper and lower envelope lines. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

on gluten hydration and dough development due to cross-linked proteins. At some level of heat treatment there is a complete lack of gluten network formation (Steertegem 2013). Hence, it is expected that the shape of the curves at 170 °C seen here (see Fig. 10) is not because of the formation of a gluten network, but is due to a different phenomenon that dominates at high temperatures. This is supported by the fact that it was not possible to wash out the gluten of these samples in a Glutomatic® System in preliminary experiments.

Fig. 11a shows mixing curves of flour samples treated at 130 °C for different times. Up to a treatment time of 15 min, the development of a gluten network could be observed. At longer times, the system destabilised as indicated by the sharp decrease in torque.

Fig. 11b shows mixing curves of flour samples treated at 170 °C for different times. For treatment times up to 3 min, a gluten network was formed, but this was mechanically destroyed after mixing times of 4–7 min. However, for treatment times between 5 min and 15 min, the rheomixer traces were stable, but the underlying mechanism will not be gluten development.

The findings show a significant impact of flour heat treatment on the hydration of protein and gluten network formation.

4. Discussion: a possible assay for heat treatment

To evaluate the success of heat treatment of flour on cake quality, a cake needs to be baked and assessed (Neill et al., 2012; Thomasson et al., 1995). The problem with testing flour with analytical methods is that various processing conditions (e.g. time-temperature combination) result in identical levels of response (i.e. RVA, rheometer, src tests). Fig. 3 shows that a peak viscosity of 2600 cP can be reached at all tested temperatures between 130 °C and 170 °C for different periods of time. However, only specific treatment conditions also result in an acceptable cake.

Here, experiments have characterised and quantified the heat treatment of flour. By combination of analytical methods, it may be possible to identify the unique time and temperature conditions at which flour was treated. Such a method is presented in Fig. 12, which plots contour lines of

i) Peak viscosity in water (see Fig. 4a), and
ii) Peak viscosity in 50% sucrose solution (see Fig. 4b)

The figure shows that there are regions of the diagram in which the contour lines cross, i.e. that a combination of the two measures can position a sample precisely on the temperature-time combination plot. This type of approach may make it possible to identify samples directly.

An unknown, heat treated flour sample can be tested and the received treatment determined. In some cases, the contour lines cross twice which means that two areas need to be considered. For example, if a flour sample exhibits a peak viscosity in water of 2650 cP and a peak viscosity in 50% sucrose solution of 1900 cP, potential heat treatments are 118 °C for 19 min or 147 °C for 2 min (see Fig. 12). Ideally, only a single solution would result. Future work should correlate these time-temperature combinations with cake quality to develop a cake flour specification. Hence, it may no longer be needed to bake a cake after the heat treatment of flour, 2 types of RVA tests might suffice.

Secondly, this type of method can help to facilitate process validation of new equipment for heat treatment. It is not always easy to measure the temperature distribution in a device and thus the temperature experienced by the product. A spiral heater (Keppler et al., 2016a, 2016b; Revtech, 2015) shall serve as an example. It consists of a helical steel pipe (length = 34.4 m) heated by the application of a low voltage directly to the pipe, through which the product is conveyed by vibrations created by off-balance motors. Factors like the temperature of the raw material, convection effects at inlet and outlet of the pipe, residence times, and thermal properties of the material affect the temperature distribution in the pipe. By using Fig. 12, only the two peak viscosities need to be measured and an equivalent treatment time and temperature can be established. The process residence time may be known approximately, making the selection of the appropriate point straightforward.

5. Conclusions

Dry heat treatment of flour for use in high ration cakes is widespread in industry. This study has first demonstrated an accurate way to heat treat flour samples and has then investigated the effect of heat

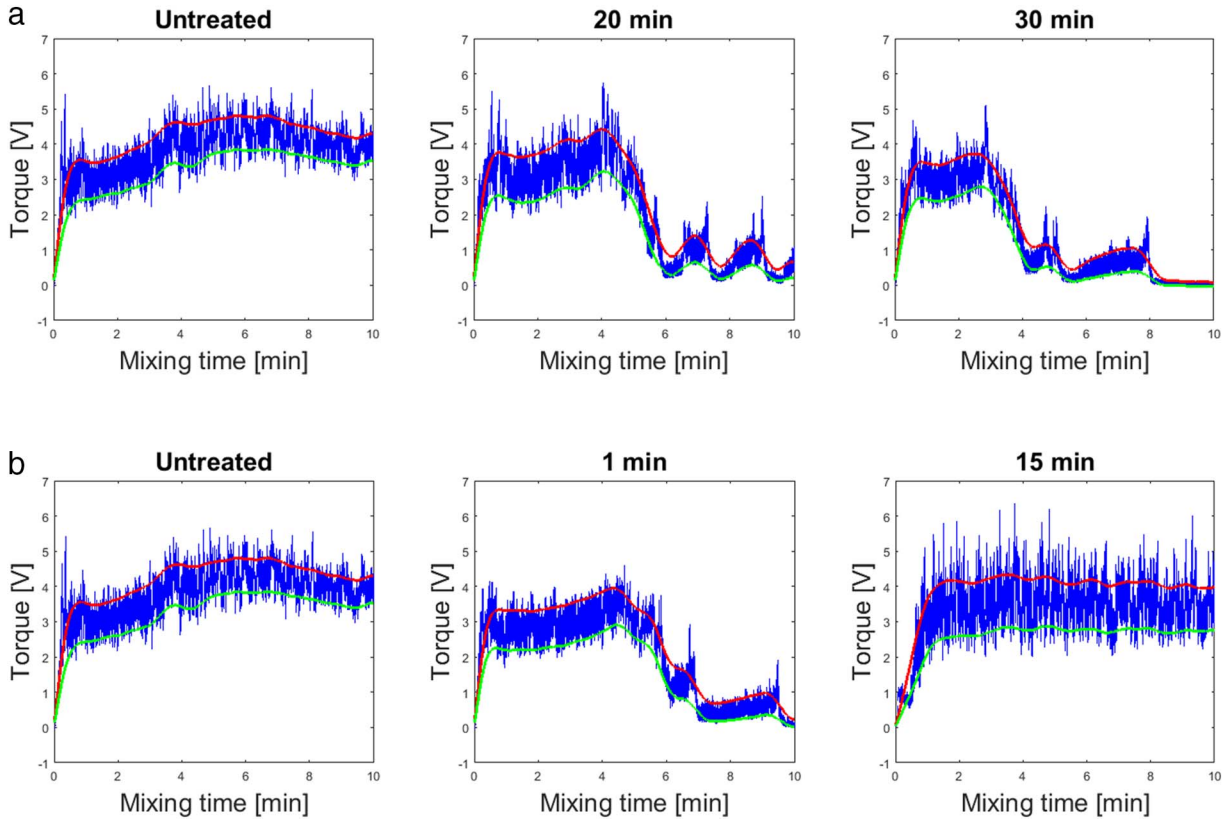

Fig. 11. Mixing curves of heat treated flour samples; red and green lines show upper and lower envelope lines a) at 130 °C b) at 170 °C.

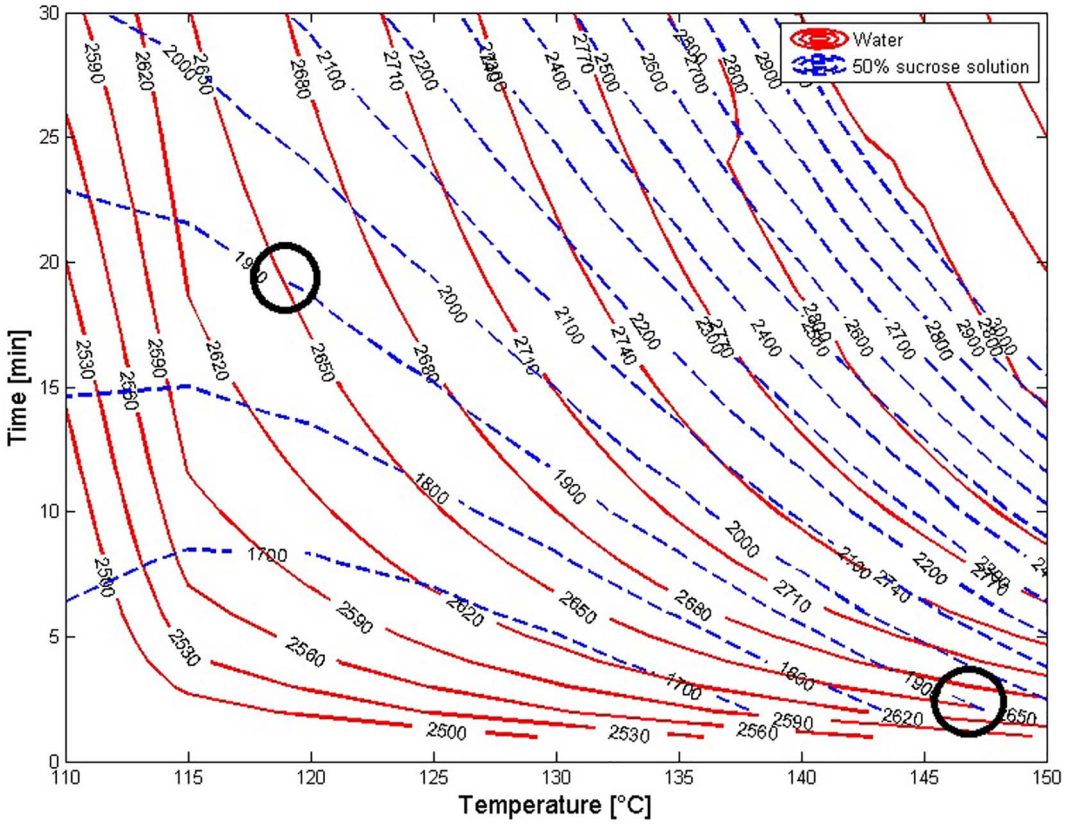

Fig. 12. Contour lines of RVA peak viscosity of heat treated flour samples in water (from Fig. 4a, shown in red in the figure) and in 50% sucrose solution (from Fig. 4b, shown in blue in the figure). The two circles show data for a heat treated flour sample with a peak viscosity in water of 2650 cP and a peak viscosity in 50% sucrose solution of 1900 cP. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

treatment on several functional aspects of flour.

Experiments in the Rapid-Visco-Analyser showed that starch granule swelling was facilitated at elevated temperatures. Results furthermore indicated improved swelling ability and increased interactions of flour polymers (in particular arabinoxylans) of heat treated flour at ambient conditions (see rheometer and SRC tests). Decreasing gluten development up to a complete lack of gluten network formation with increasing intensity of heat treatment was found using a rheomixer.

The study has demonstrated that two types of RVA tests generate sets of results that cross, so that a combination of the two can position a sample on the temperature-time combination plot. This method suggests that it may not be necessary to bake a cake to evaluate the outcome of a heat treatment process. However, it will be desirable to find analytical methods that provide contour lines that are dissimilar and ideally only cross once. Furthermore, the method would need to be repeated for different flour batches. However, if the shape of a surface is known, only a small experimental design would be needed. Future work is necessary to correlate fully the identified time-temperature conditions at which flour was treated with the final cake quality attributes. This might enable the development of a cake flour specification.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.foodres.2018.02.041.

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